

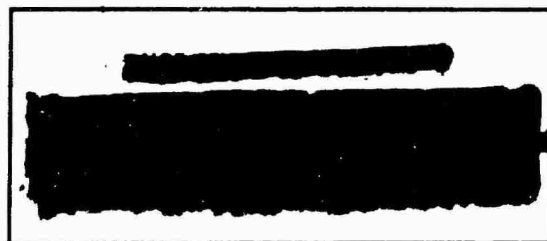
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EVALUATION TEST METHODS  
for  
REFRACTORY METAL SHEET MATERIALS



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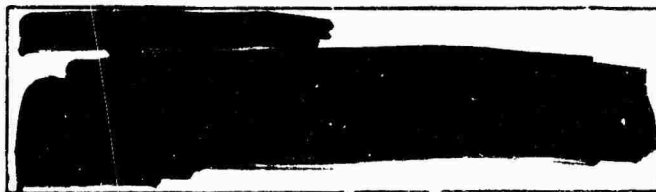
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Supersedes MAB-192-M

Report of the  
TEST METHODS SUBPANEL  
REFRACTORY METALS SHEET ROLLING PANEL

EVALUATION TEST METHODS  
FOR  
REFRACTORY METAL SHEET MATERIALS

Prepared By The  
MATERIALS ADVISORY BOARD  
Division of Engineering and Industrial Research  
National Research Council

as a service of  
The National Academy of Sciences  
to the  
Office of Defense Research and Engineering  
Department of Defense



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## PREFACE

This is the third revision of recommended test methods for refractory metal sheet published by the Materials Advisory Board Refractory Metals Sheet Rolling Panel. The Panel's objective was to provide a standard basis for the test evaluation of sheet materials produced under various Department of Defense development programs. A subpanel was appointed for this purpose. After a thorough canvas of prominent suppliers and users of refractory sheet metals, the first recommendations were published in September 1961 as MAB Report 176-M to define common tensile, creep-rupture, bend, and thermal properties tests. Subsequently, as the Department of Defense programs progressed, more specialized tests became necessary to evaluate fabrication characteristics and to provide preliminary design data. Again, after discussion with principal organizations engaged in direct applications, a revised edition was published April 22, 1963, as MAB-192-M. Since that time, criticisms and comments have been reviewed by the subpanel with qualified individuals representing governmental and commercial laboratories. The present edition is based upon these reviews. Changes have been made in the tests described in Sections 1 through 6. The tests in Sections 7 through 15 are unchanged from Report 192-M.

"Standard" methods, by definition, cannot solve the many special design and fabrication problems associated with particular equipment, processes, or applications. These procedures are not intended to meet unique situations. When used properly, however, standardization should improve the validity of comparisons of data reported by different laboratories. It is particularly important for laboratories to report clearly detailed methods and deviations used in conducting tests.

In preparing these recommendations, the subpanel has deferred wherever possible to prominent ASTM standards. The subpanel thanks the many government and industrial laboratories who gave valuable advice and comment.



EVALUATION TEST METHODS  
FOR  
REFRACTORY METAL SHEET MATERIALS

1. Tensile Test, Room Temperature and Elevated Temperature

1.1 Specimen Dimensions

The specimen size recommended is intended particularly for preliminary evaluation of small pilot lots of new alloys. The specimen is somewhat smaller than the standard ASTM specimen. The smaller size provides several advantages: 1) requires less material, 2) allows more rapid heating, 3) improves gage temperature uniformity, and 4) reduces grip loads. For test evaluation of production lots, particularly against firm purchase order specifications, the standard ASTM specimen is satisfactory unless the laboratory performing the tests can demonstrate that the specimen size does not affect test results. If this is shown, the subsize specimen is acceptable for production lot testing.

1.1.1 Subsize Specimen Dimensions Shall be as Follows:

Gage length -  $1.00 \pm 0.003$  inch

Gage width - The gage width limits shall be  $.250 \pm .005$  inch with a maximum permissible variation in a single uniform width specimen of .001 inch. If desired, the width at the center may be reduced by not more than .005 inch to favor a center fracture. In this case, the width must be uniformly tapered from both ends to the center, the end widths may not differ by more than .001 inch, and the entire gage section must be within the  $.250 \pm .005$  limits. The most important consideration is accuracy of measurement of width and thickness. An accuracy of .2 per cent should be assured and sufficient measurements should be made to clearly establish the position and exact dimensions of the minimum cross section.

Gage thickness - sheet thickness

Fillet radius -  $0.250 \pm 0.062$  inch  
- 0.000

(A larger fillet radius may be used, if required for exceptionally brittle materials. Deviations in dimensions should be clearly noted in reporting test results.)

Grip section - optional

1.2 Gripping Method - Optional

1.3 Surface Finish

The specimen gage section surface finish shall be the same as the

finished sheet. No special surface treatments shall be used which will affect the surface condition of the specimen as compared with the final sheet surface. The edges of the gage section shall be carefully deburred and polished longitudinally with 00 emery or equivalent.

#### 1.4 Pretest Inspection

The sheet material should be checked carefully for surface contamination by microhardness tests and metallographic inspection of cross sections. Chemical analyses of the 0.005 to 0.01 inch surface layer for oxygen, nitrogen, hydrogen, and carbon can be made on chips; but to avoid spurious results because of the increased surface area and high temperature generated during chip formation, most laboratories prefer the use of solid samples (comparing full cross-section analysis with core analysis). To supplement chemical and metallurgical inspection, specimens of columbium- and tantalum-base alloys may be precision weighed to the nearest 0.1 mg after: (a) machining and cleaning, (b) thermal treatment, and (c) test. This procedure is useful in the detection of serious contamination. The specimens should be checked for edge cracks prior to test by low-power optical inspection.

#### 1.5 Loading Apparatus and Methods

The test machine shall comply with the requirements of ASTM E-4 and specimen alignment shall meet the requirements of ASTM E-21.

#### 1.6 Strain Measurement & Strain Rate for Tests, up to and including 1500F

##### 1.6.1 Method

A recording extensometer shall be used to determine strain from zero to a minimum of 0.6 per cent offset. Calibration, attachment, and accuracy shall comply with the requirements of ASTM E-83 and E-21. For offset yield strength determination, a class C or better extensometer shall be used; for modulus determinations, a class B-1 or better extensometer shall be used. For strain measurement beyond the 0.6 per cent offset strain, it is permissible to use gage marks in lieu of extensometer measurement. When a modulus determination is made, the plastic strain shall be limited to less than 0.1%. Interruption of tests is permissible to exchange extensometers or to change strain rates after modulus determination.

##### 1.6.2 Strain Rate

A strain rate of  $0.05 \pm .01$  in/in/min shall be used from zero strain to fracture. Where strain rate is not measured directly, the method of determination shall be reported. For modulus determinations, or for the testing of brittle materials, a slower strain rate should be used up to 0.1% plastic strain:  $0.005 \pm .002$  in/in/min is recommended.

## 1.7 Strain Measurement and Strain Rate for Tests Above 1500F

### 1.7.1 Method

It is recognized that recording extensometers for service in vacuum or inert gas chambers at temperatures above 1500F may not be widely available. Strain recording for tests at temperature where an extensometer is not available may be made by controlling the cross-head speed, preferably by means of a pacer, to provide a controlled rate of head separation in inches per minute. Where strain rate is approximated by controlling crosshead speed, speed shall be measured under actual loading conditions.

### 1.7.2 Strain Rate

A strain rate of  $0.05 \pm .01$  in/in/min shall be maintained from zero strain to fracture. For modulus determinations, a slower strain rate may be used up to 0.1% plastic strain:  $0.005 \pm .002$  in/in/min is recommended.

## 1.8 Room Temperature Control

All tensile testing shall be conducted in a room whose ambient temperature shall be held within a 65F to 85F range. Actual temperature shall be reported.

## 1.9 Heating for Elevated Temperature Tests

### 1.9.1 Temperature Control

The indicated temperature at any point along the gage length of the specimen shall not vary by more than the following from the nominal test temperature:

<u>Nominal Test Temperature</u>	<u>Maximum Permissible Variation</u>
Up to and including 1800F	$\pm 10^{\circ}$
Above 1800F up to and including 2800F	$\pm 20^{\circ}$
Above 2800F up to and including 3500F	$\pm 30^{\circ}$
Above 3500F up to and including 4000F	$\pm 35^{\circ}$

### 1.9.2 Temperature Measurement

Base metal couples may be used for test temperatures up to 2100F and noble metal couples to as high as 2800F. The recommendations of ASTM E 26-58T with regard to temperature measurement should be followed wherever applicable. This is particularly important with regard to calibration, checking, re-use, and attachment of thermocouples and the calibration of instruments.

For test temperatures in the 2500F to 4500F range, high-temperature thermocouples or optical pyrometry may be used. Data must be provided to demonstrate the accuracy of the temperature sensor device.

1.9.3 Heating Rate

The specimen shall be brought to test temperature in no less than five minutes or no more than 60 minutes, and held at test temperature for 15 minutes before loading. Complete information on heating rate and soaking time must be reported with the results.

1.10 Test Environment

All elevated temperature testing shall be conducted under conditions to ensure that the surface of the specimen will not be chemically affected during the test. Low pressure, inert gas, getters, or coatings, or combinations of any of these may be used, but in any case proof must be provided to show clearly that the specimen surface was essentially unaffected by the test condition.

1.11 Post-test Inspection (Elevated temperature tests)

Following test, a representative specimen or specimens shall be subjected to the same tests as specified in Section 1.4. Any changes in chemical analyses, hardness, or structure shall be noted.

1.12 Reports

Test reports shall include material history, test methods, and results. Elongation measurements shall be made in accordance with the methods of ASTM-E21. Complete data should be reported on a simple form such as shown in Appendix A.

2. Compression Test

2.1 Scope

This procedure applies to the compression testing of refractory metal and alloy sheet at room temperature to determine:

- a. Compressive yield strength
- b. Compressive modulus

2.2 Equipment and Materials

2.2.1 Testing Machine

All tests shall be made on a universal testing machine or equivalent which complies with the requirements of ASTM E-4.

### 2.2.2 Test Fixture

Specimens shall be tested in a fixture that complies with the requirements of ASTM E9-61. The fixture shall provide lateral support against buckling and must hold the specimen so that the load is applied axially and uniformly. Both ends of bearing blocks must bear on smooth finished plane surfaces that are parallel. Bearing blocks shall not suffer appreciable deformation. The jig must permit the specimen to deform and should be lubricated on all bearing surfaces to minimize friction.

### 2.2.3 Extensometer

Extensometers shall conform with the requirements of ASTM E-83.

## 2.3 Test Specimens

### 2.3.1 Blank Dimensions

The specimen size is dictated by the type of test fixture (ASTM E9-61). For the Sandorff-Dillon jig, the specimen shall be 0.5 inch wide by 3.1 inch long with a 2.0 inch gage length. The specimens shall be flat and of full thickness.

### 2.3.2 Blank Preparation

Test blanks of the correct orientation shall be taken from the sheet. Test surface shall be representative of the sheet surface. The edges of the specimen shall be deburred and polished longitudinally with 00 abrasive. Specimens shall be checked for soundness and edge cracks prior to testing. Low power optical inspection is suggested.

The thickness and width of all specimens shall be determined by a minimum of 3 readings within the gage length. Ends of the specimen shall be parallel to within 0.0005 inch based on measurements of the length of the specimen from one edge to the other. (From ARTC-13)

## 2.4 Test Procedures

### 2.4.1 Method

All tests shall be conducted in accordance with ASTM E-9-61, Compression Testing of Metallic Materials.

### 2.4.2 Strain Rate

A strain rate of  $0.005 \pm 0.002$  in/in/min shall be used for all tests. Strain rates may be approximated by means of a strain pacer based on crosshead movement.

#### 2.4.3 Test Temperature

All tests shall be conducted at 65-85F unless otherwise specified.

#### 2.5 Report Requirements

- a. Material - condition, history, and specimen orientation
- b. Test temperature and strain rate
- c. Compressive yield strength at 0.2 per cent offset
- d. Compressive modulus
- e. Results of pre-test inspection

A recommended test form is shown in Appendix A.

#### 2.6 References

J. R. Kattus and H. L. Lessley, "Determination of Compressive, Bearing and Shear Creep of Sheet Metals," Proc. ASTM, Vol. 61, 1961, p. 920-930.

Fourth Report of Task Force on Uniform Procedures for Structural Design Data Collection of the Panel on Titanium Sheet Rolling Program of the MAB, "Uniform Testing Procedures for Sheet Materials, Part 3".

W. S. Hyler, "An Evaluation of Compression Testing Techniques for Determining Elevated-Temperature Properties of Titanium Sheet," Battelle Memorial Institute, TML Report No. 43, June 8, 1956.

G. Gerard, "Results of Tests Evaluating Compression Testing Techniques of Sheet Materials at Elevated Temperature," National Academy of Sciences, MAB Report MAB-122-M, 1958.

#### 3. Notched Tensile Test

The purpose of this test is to determine the notch-strength ratio of refractory metal alloy sheet in the fabricating temperature range.

##### 3.1 Specimen Dimensions and Gripping Methods

The exact geometry of the notch in the notched tensile test must be determined on the basis of a number of variables--base material, coating system, and overall purpose of the test. Where it is intended to serve as an empirical evaluation of fabricability on materials of limited ductility, a somewhat generous notch radius may be used. The geometry of this edge-notch specimen is as follows: 1) gage section 0.050-in. wide by 1.0 in. long; 2) edge notch at center of gage section with notch depth of 0.125 in. on each side; 3) notch root radius - .0045 to .0055 in.;  $K_t = 5.8$ ;

and 4) flank angle of  $60^{\circ}$ . Any suitable grips which minimize the loading eccentricity to 0.001 in. or 15% elastic strain, whichever is less, may be used. Pin loading is preferred with machining tolerance given with respect to pin holes. A recommended specimen is shown in Fig. 3.1.

For more ductile materials, where either fabrication or design information is sought, a precracked specimen should be used. Such a notch has not yet been standardized, but preliminary efforts have been made by ASTM Committee E-24 on Fracture Testing of Metals. Pending an ASTM standard, and to provide for a uniform amount of cold worked metal in the vicinity of the crack, the stress used in generating the fatigue crack should be limited to 20% of the yield strength.

### 3.2 Testing Machine Requirements

The testing machine shall conform to requirements given in Sec. 1.

### 3.3 Testing Conditions

The crosshead speed shall be maintained with the range of 0.005 to 0.01 in/min.

Tests shall be conducted at room temperature and at additional temperatures starting at 200F, and increasing in 100F increments until a notch strength ratio of unity has been exceeded. Indicated control shall be within  $\pm 5F$  of nominal test temperature.

### 3.4 Test Results and Report

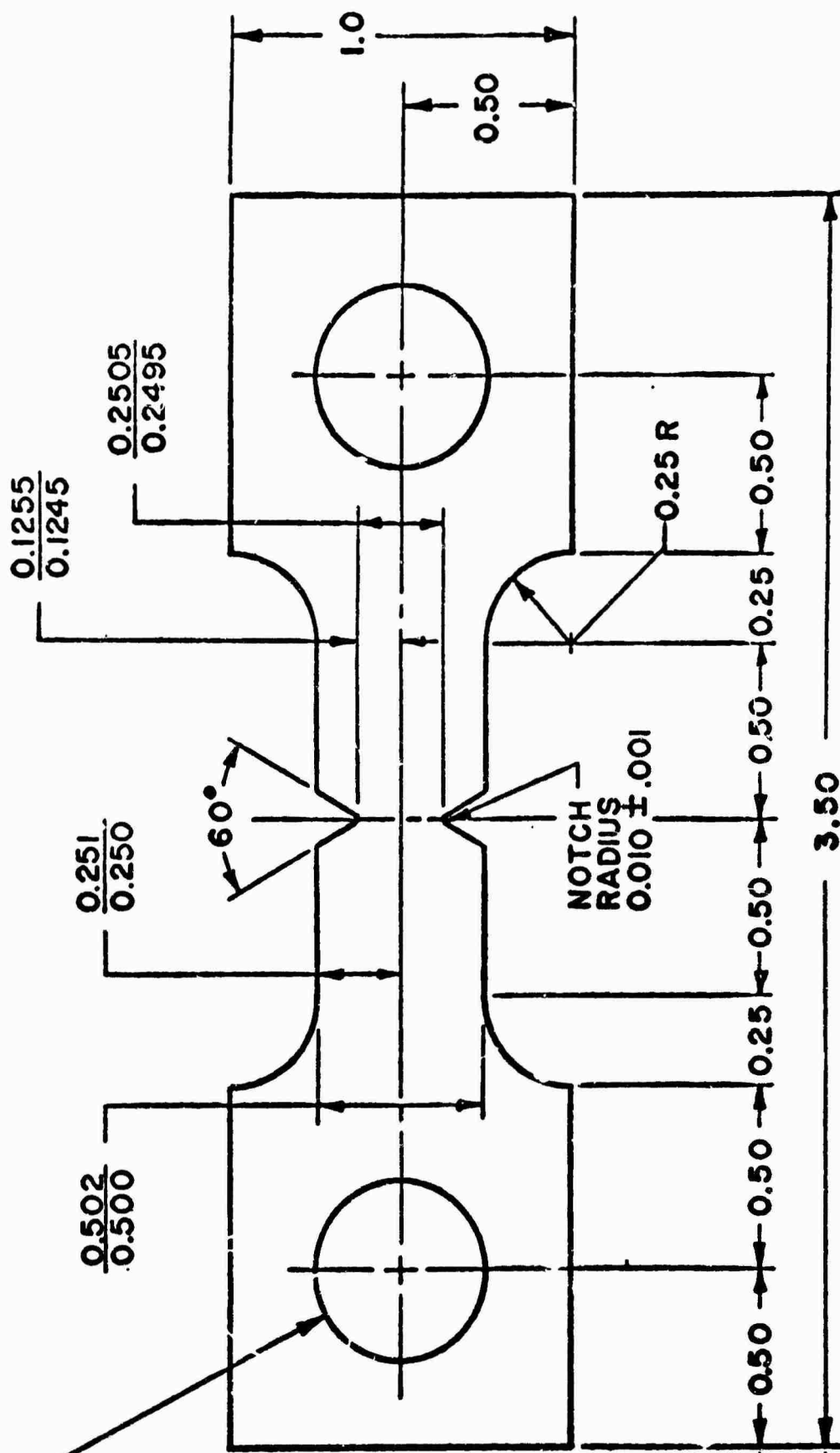
The notch strength is the maximum load divided by the net cross-sectional area at the root of the notch. A value of the ultimate tensile strength of an unnotched specimen shall be determined for the same temperatures from the sheet material, as indicated in Sec. 1. The notch strength ratio at a given temperature is the notch strength divided by the ultimate strength of an unnotched specimen.

All pertinent test data shall be reported including test temperature, loading rate, full details on chemical and metallurgical features of the stock, and appropriate strength data.

### 3.5 References

F. A. McClintock, "On Notch Sensitivity," Welding Journal, Vol. 40, May 1961, p. 202-209 S.

J. F. Watson & J. L. Christian, "Low Temperature Properties of K-Monel, Inconel-X, René 41, Haynes 25 and Hastelloy B Sheet Alloys," Trans. ASME, Journal of Basic Engineering, June 1962, p. 265-277.



**NOTCH TENSILE SPECIMEN  
(DOUBLE SIZE)**

**Fig. 3.1**



#### 4. Shear Ultimate Strength Test

The purpose of this test is to determine the maximum shear that a sheet material can withstand in a structural element such as a shear web. This test will also evaluate the influence of directionality.

##### 4.1 Specimen and Gripping Methods

The shear test section is an axial shear path of 3/16 inch, which is defined by two 1/16 inch holes spaced 1/4 inch apart on the axis and two offset slots at 45° to the axis, Fig. 4.1.1. Axiality of the load is insured by loading through pin holes which are also on the axis of the shear path. The recommended specimen is shown in Fig. 4.1.1 a. The clevises at the pin holes should be tight to prevent lateral displacement or buckling of the heads. If buckling is encountered in testing thin sheet, the 0.063 inch holes may be relocated off the center line by .015 inch to reduce the bending moment. Specimen modifications shall be reported in detail.

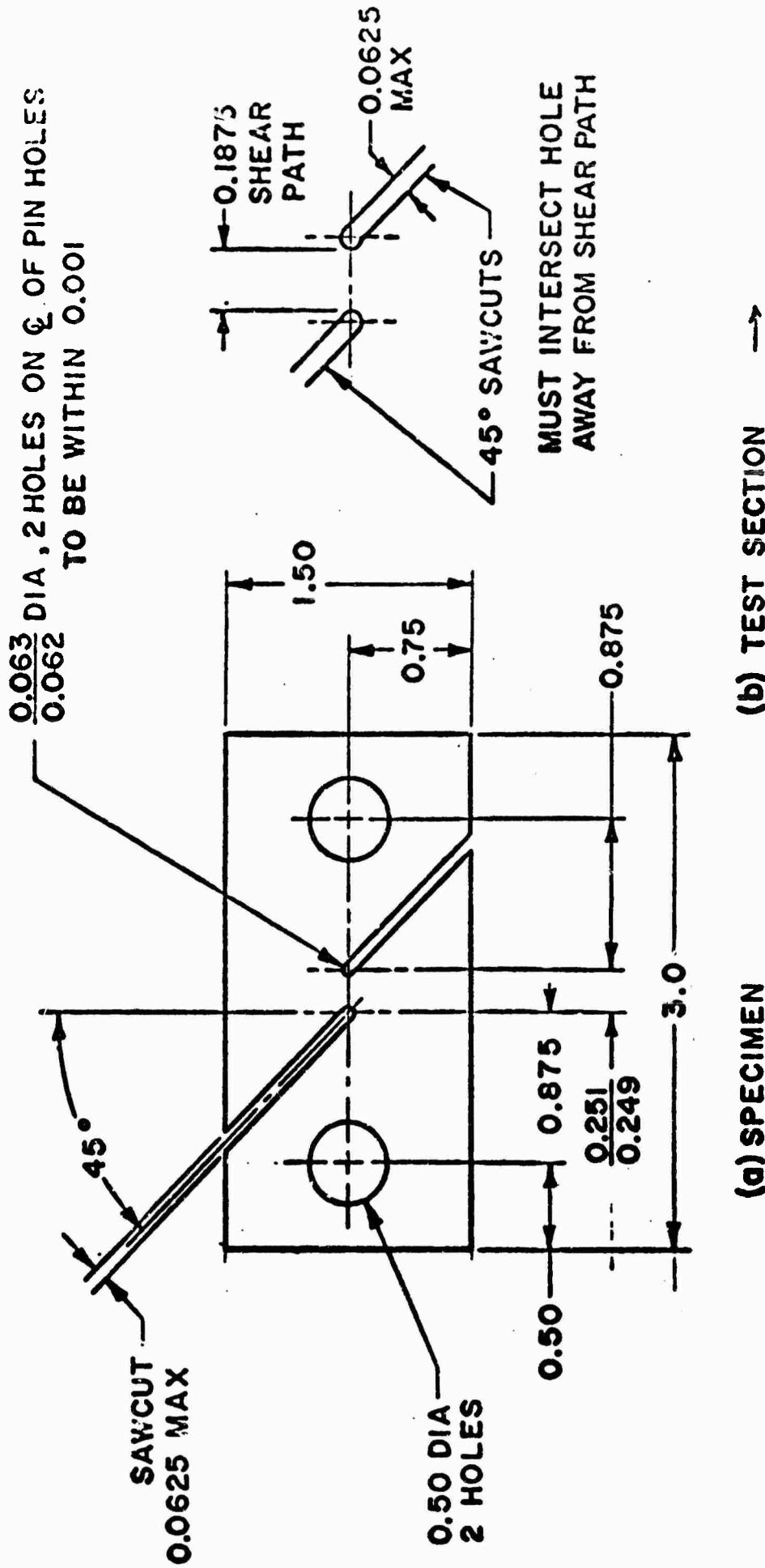
- 4.1.1 The holes at the ends of the shear path must be smooth. The saw cuts shall be made so that they intersect the hole without cutting the shear path. Therefore, they shall be kept on the side of the hole away from the shear path, Fig. 4.1.1. The direction of the axis of the shear path shall be accurately determined with respect to the rolling direction.
- 4.1.2 The thickness of the specimen shall be determined using a micrometer reading within the test section. The shear path or distance between the hole edges along the centerline, on both sides of the sheet, shall be determined within 0.001 inch by a standard instrument, such as an optical comparator.
- 4.1.3 The testing machine shall conform to the requirements of ASTM E-4. The loads used in testing shall be within the loading range of the testing machine, or defined by ASTM E-4, and the specimen alignment shall meet the requirements of ASTM E-21.

##### 4.2 Test Procedure

The crosshead speed shall be maintained within the range of 0.005 to 0.01 in/min. An extensometer is not required, but adequate means must be provided to determine the maximum shear load.

##### 4.3 Test Results and Report

The ultimate shear strength is the maximum shear load divided by the net shear path area. The fracture shall be inspected to determine if buckling or any other irregularities were influencing the test.



ALL DIMENSIONS IN INCHES

SHEAR SPECIMEN

FIG. 4.1.1

All pertinent data shall be reported, including specimen orientation with respect to rolling direction, test temperature, loading rate, full details on chemical and metallurgical features of the stock, and strength data.

#### 4.4 References

H. E. Davis, G. E. Troxell, and C. T. Wiskocil, "The Testing and Inspection of Engineering Materials," McGraw-Hill Book Co., Inc., New York, 1949, p. 113.

R. W. Fenn, Jr. and R. W. Clapper, "Evaluation of Test Variables in the Determination of Shear Strength," Proc. ASTM, Vol. 56, 1956, p. 842-858.

W. W. Breindel, C. L. Seale, and R. L. Carlson, "Evaluation of a Single-Shear Specimen for Sheet Material," Proc. ASTM, Vol. 58, 1958, pp. 862-868.

J. R. Kattus and H. L. Lessley, "Determination of Compressive, Bearing, and Shear Creep of Sheet Metals," Proc. ASTM, Vol. 61, 1961, pp. 920-930.

Fourth Report of Task Force on Uniform Procedures for Structural Design Data Collection of the Panel on the Department of Defense Titanium Alloy Sheet Rolling Program of the Materials Advisory Board, "Uniform Test Procedures for Sheet Materials," Part 5: Shear Test.

#### 5. Bend Test

##### 5.1 Scope

This test determines the ability of refractory metal and alloy sheet to withstand a 90 to 105° bend over a radius of curvature up to and including 10 times the sheet thickness (10t). It is applicable to testing sheet ranging in thickness from 0.010 to 0.100 inches over a wide temperature range.

This test is designed to determine:

- a. Ductile to brittle transition temperature for a given bend radius;
- b. Minimum bend radius for a given test temperature.

The test is intended to provide a semi-quantitative measure of ductility and sheet quality. It should be noted that data obtained from tests by this procedure have not been correlated directly with other properties or performance data. The tests results are not intended to serve as an absolute measure of ductility and/or formability, and should not be used as structural or process design parameters.

## 5.2 Test Equipment and Fixtures

### 5.2.1 Testing Machine

All bend tests shall be made on a universal testing machine, power brake, or other hydraulic or motor driven loading devices in which the rate of crosshead or punch travel can be controlled at a constant known value during the test.

### 5.2.2 Bend Fixture

Tests shall be made in a rigid fixture which will allow the specimen to bend as a simple beam in three-point loading. The bend fixture shall be a channel die with a geometry as indicated in Fig. 5.2.1 and Table 5.2.1.

The support span (S) is taken as the horizontal distance between the vertical faces of the channel die (Fig. 5.2.2). The span is a function of nominal sheet thickness as shown in Figure 5.2.2 and is designed to permit bends of up to and including  $10t$  radius for each sheet thickness range. The die may be made adjustable to provide for variable spans, or an individual die may be made for each span required. The specimen supports shall be rigid and must not deflect during loading.

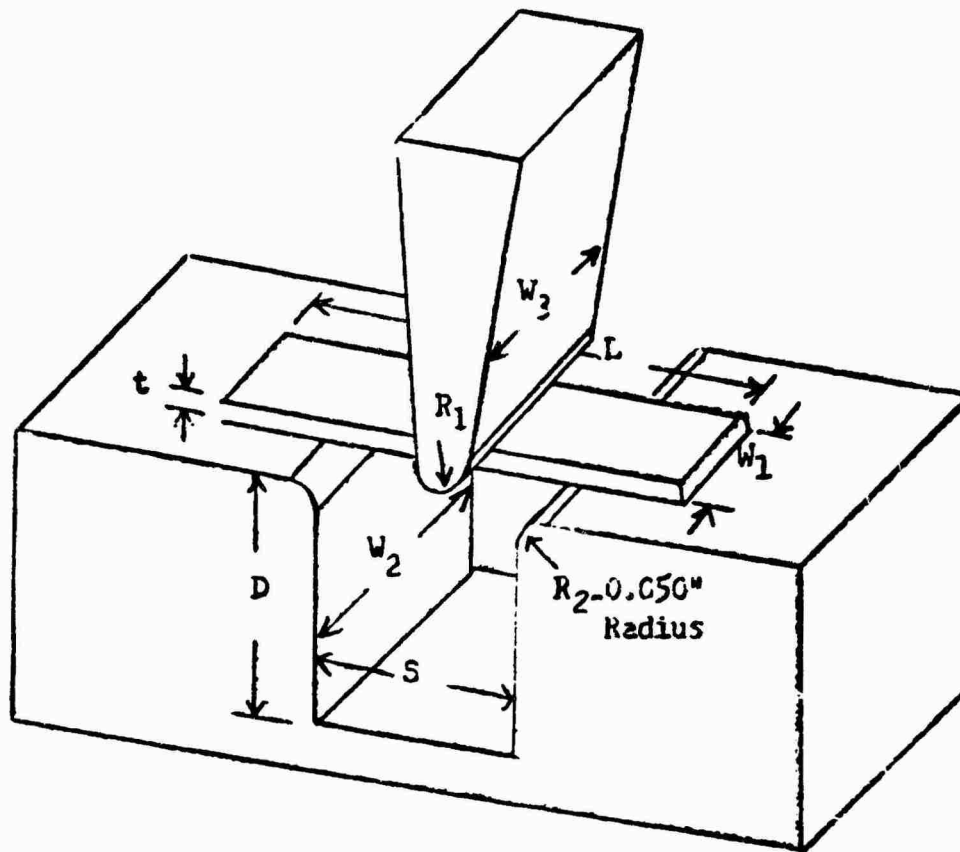
The shoulders on which the test specimen bears shall have a radius of 0.050 inch for all test fixtures. They shall be made of hardened steel with a polished surface. The specimen supports may be an integral part of the test fixture or may be replaceable pins or rollers.

Care should be taken in placing the specimen on the fixture to ensure that the axis of bend will be perpendicular to the specimen edges.

The depth (D) of the test fixture shall be equal to or greater than the span (S). The minimum width of the test fixture ( $W_2$ ) at the point of specimen support and of the punch ( $W_3$ ) is dependent on sheet thickness. There is no limit on maximum depth and width of the test fixture and punch.

The punch of radius  $R_1$  shall be made of hardened steel with a polished surface. The radius ( $R_1$ ) is a whole number multiple of the nominal sheet thickness ( $1t$ ,  $2t$ ,  $3t$ , etc.). The radius may be machined as an integral part of the punch or may be formed by a replaceable pin or rod. The punch must be rigid and should not twist or deflect horizontally during loading.

## 5.3 Test Specimens



NOMINAL SHEET THICKNESS -in.	FIXTURE			TEST SPECIMEN			
				MINIMUM		SUGGESTED	
	S	D*	$W_2 * W_3$	$W_1$ *	L*	$W_1$	L
	in.	in.	in.	in.	in.	in.	in.
Over 0.089 to 0.030 inc.	1.0	1.0	1.0	0.5	2.0	0.5	2.5
Over 0.030 to 0.050 inc.	1.25	1.25	1.0	0.5	2.25	0.5	2.5
Over 0.050 to 0.065 inc.	1.50	1.50	1.25	0.75	2.75	0.75	3.25
Over 0.065 to 0.075 inc.	1.75	1.75	1.25	0.75	3.00	0.75	3.25
Over 0.075 to 0.085 inc.	2.00	2.00	1.50	1.00	3.50	1.00	4.25
Over 0.085 to 0.095 inc.	2.25	2.25	1.50	1.00	3.75	1.00	4.25
Over 0.095 to 0.105 inc.	2.50	2.50	1.50	1.00	4.00	1.00	4.25

\* Minimum Value

Load Rate = 1.0 in/min Punch Travel

$R_1 = 10t$  Max.

Figure 5.2.2

BASIC CONCEPTS OF BEND TEST FOR REFRACTORY METAL SHEET

### 5.3.1 Blank Dimensions

The minimum dimensions of the test blank are determined by sheet thickness as indicated Table 5.3.1. The specimen width ( $W_1$ ) should not exceed the actual width of the test fixture ( $W_2$ ) or of the punch ( $W_3$ ). There is no maximum limit on specimen length. In order to facilitate specimen preparation and simplify test procedures and to further standardize procedures, three basic blank dimensions are suggested, as shown in Table 5.3.1.

Table 5.3.1

#### Test Specimen Dimensions

Nominal Sheet Thickness (t)	Minimum Dimensions		Suggested Dimensions	
	Width ( $W_1$ )	Length ( $W_2$ )	Width ( $W_1$ )	Length ( $W_2$ )
over .009 to .030 incl.	0.50	2.00	0.50	2.50
over .030 to .050 incl.	0.50	2.25	0.50	2.50
over .050 to .065 incl.	0.75	2.75	0.75	3.25
over .065 to .075 incl.	0.75	3.00	0.75	3.25
over .075 to .085 incl.	1.00	3.50	1.00	4.25
over .085 to .095 incl.	1.00	3.75	1.00	4.25
over .095 to .105 incl.	1.00	4.00	1.00	4.25

### 5.3.2 Blank Preparation

Test blanks shall be taken from the sheet with required orientation. The test specimen surface must be fully representative of the finished sheet surface and no processing of the blank which may alter surface condition is permitted. Welded specimens, however, should be ground or milled to provide a flat surface at the point of punch and test fixture contact.

Edges should be filed, ground, or milled to remove burrs and defects. Sharp edges shall be broken or rounded by polishing with 120 grit abrasive in a longitudinal direction. Care must be taken to avoid excessive rounding of the edges. Edges should be free of cracks or notches that may initiate fracture.

The thickness and width of all specimens shall be measured in the region where bending will occur.

### 5.4 Test Procedures

#### 5.4.1 Test Temperature

Room temperature tests shall be made between 65 and 85F; however, the actual temperature must be within  $\pm 5^\circ$  of the reported temperature.

Temperatures above or below ambient shall be controlled to within  $\pm 5^\circ\text{F}$  of the reported temperature. Temperature shall be measured with a calibrated thermocouple contacting the specimen except for tests at room temperature or in constant temperature baths, where precision thermometers may be used. Specimens shall be soaked for 3 to 5 minutes after reaching temperature before loading. The punch and test fixture shall be maintained at the same temperature as the test specimen.

#### 5.4.2 Test Atmosphere

Tests may be conducted in air or any other liquid or gaseous medium providing the surface condition is not materially altered during the test.

#### 5.4.3 Load Rate and Testing

All tests shall be conducted at a constant rate of crosshead or punch travel of 1.0 inches per minute. The specimen shall be bent beyond  $90^\circ$  angle a sufficient amount to produce a free bend angle of  $90^\circ$  to  $105^\circ$  after springback on unloading. If the sample fails by cracking before completing the bend, the test shall be stopped immediately. The final bend angle after successful bending or fracture shall be measured to the nearest  $5^\circ$ .

#### 5.4.4 Inspection and Failure Criteria

A sample shall be judged to have failed if cracks develop on the tensile side or edges or if delamination occurs. Inspection for detecting failure shall be consistent with practices already established for aluminum, steel and titanium. The sensitivity for revealing cracks need not be finer than the characteristic of dye penetrant techniques. To facilitate visual detection, examination at a magnification of at least 10 diameter is suggested.

### 5.5 Test Results

#### 5.5.1 Bend Transition Temperature

The transition temperature range is defined as the lowest temperature, for a given bend radius, at which a sound  $90^\circ$  to  $105^\circ$  bend can be produced and the highest temperature in which specimen failure occurs. It shall be determined to the nearest  $25^\circ\text{F}$ . To facilitate comparison and minimize quantity

of tests, it is suggested that initial tests be conducted with a  $4t$  bend radius at  $100^\circ\text{F}$  intervals to bracket the transition temperature.

#### 5.5.2 Minimum Bend Radius

The minimum bend radius at ambient (65 to  $85^\circ\text{F}$ ) or any other specified temperature is defined as the minimum punch radius ( $R_1$ ) which will produce a sound  $90$  to  $105^\circ$  bend. Minimum bend radius shall be determined to the nearest integer value of thickness ( $1t$ ,  $2t$ ,  $3t$ , etc.).

To minimize the number of tests, it is suggested that initially tests be conducted at even interger values of  $t$  ( $2t$ ,  $4t$ ,  $6t$ , etc.) to bracket the minimum bend radius.

#### 5.5.3 Bend Angle and Springback

The final bend angle after testing shall be measured to the nearest  $5^\circ$ . The springback angle shall be calculated by subtracting the final bend angle from the angle under load at the end of the test. The angle under load may be measured directly or may be calculated from the bend fixture geometry (geometrical considerations) and the distance of punch travel.

#### 5.6 Report Requirements

The following data shall be reported:

- a. Material, condition, and history (including welding or coating procedures, if any). Length, width, and thickness of test specimen and length of test fixture span.
- b. Test temperature, and rate of crosshead or ram travel.
- c. Minimum successful and next lower failed punch radius ( $R_1$ ) for base bend ductility at a given temperature.
- d. The lowest temperature at which a successful test was achieved and the highest temperature at which failure was encountered.
- e. Springback angle as a function of bend radius and test temperature.



The recommended report form is shown in Appendix A.

6. Bearing Strength Test

6.1 Test Specimen

6.1.1 The specimen blank shall be cut from a flat sheet with the required orientation with the rolling direction. The blanks shall be 1.25 inches minimum width. The bearing holes shall be drilled and reamed as shown in Fig. 6.1.2. The edges shall be deburred with No. 00 emery paper, without reducing the thickness below that of the sheet.

6.1.2 The loading hole shall be large enough to prevent any plastic deflection during the test. A recommended specimen is shown in Fig. 6.1.2. In testing soft materials, the specimen may require modification to prevent premature failure in the loading hole. Also, in testing thin sheet, some adjustment may be needed to keep the  $\frac{D_B}{T}$  ratio between 5 and 8 while maintaining an edge

distance (E) equal to at least 1.5 times the bearing hole ( $D_B$ ) diameter or  $E \geq 1.5 D_B$ . This is essential to avoid shearing of the hole. In any case, the specimen details should be reported for all tests particularly since the apparent bearing strength increases with increasing  $\frac{E}{D_B}$ . A suggested alternate specimen is shown in Fig. 6.2.2.

6.2 Loading

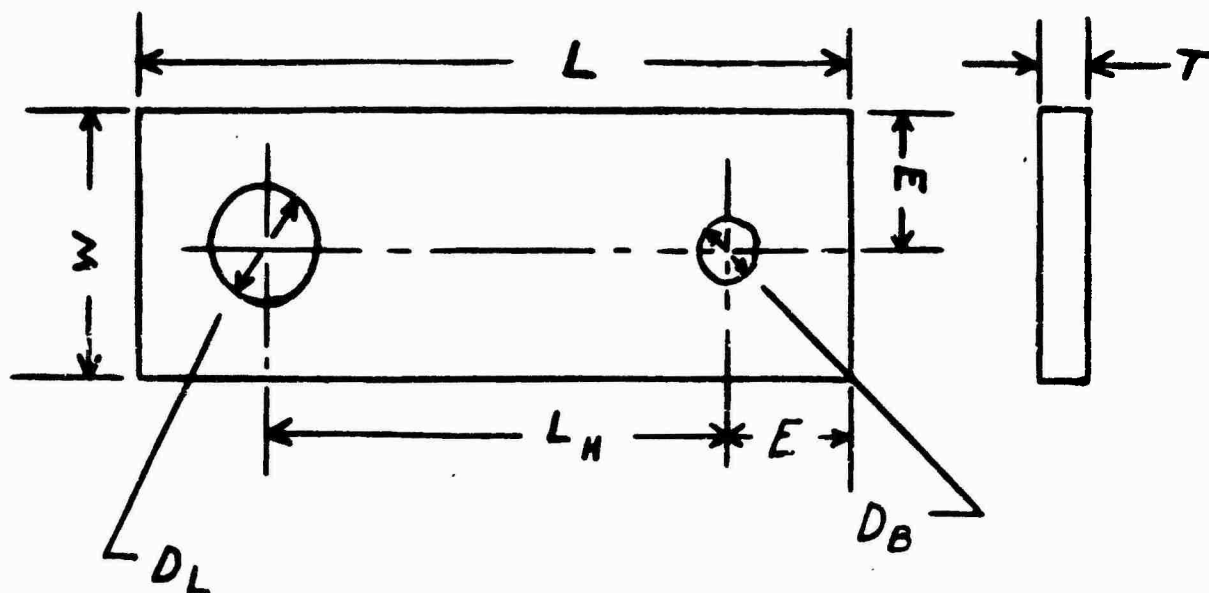
6.2.1 The testing machine shall conform to the requirements given for tension tests, Section 1.

6.2.2 Clevis grips and pins for the bearing hole and loading hole must provide sufficient lateral constraint to prevent buckling of sheets without imposing undue frictional effects, or preventing lateral expansion of the bearing surface.

6.2.3 Not more than five (5) pounds should be required to pull the specimen out of the clevis before the pin is inserted.

6.2.4 Total diametric clearance between the bearing pin and bearing hole shall not exceed 0.0005 inch.

6.2.5 The bearing stress is defined as the load divided by the product of the specimen thickness, and bearing hole diameter.



DIMENSIONS

	Inches	
Length	$L$	$3.50 \begin{smallmatrix} + 0.10 \\ - 0.00 \end{smallmatrix}$
Width	$W$	$1.25 \begin{smallmatrix} + 0.05 \\ - 0.00 \end{smallmatrix}$
Thickness	$T$	Sheet Thickness
Edge Distance	$E$	$0.625 \begin{smallmatrix} + 0.05 \\ - 0.00 \end{smallmatrix}$
Bearing Hole	$D_B$	$0.3125 \begin{smallmatrix} + 0.0005 \\ - 0.0000 \end{smallmatrix}$
Loading Hole	$D_L$	$0.5005 \begin{smallmatrix} + 0.0005 \\ - 0.0000 \end{smallmatrix}$
Center to Center	$L_H$	$2.25 \begin{smallmatrix} + 0.10 \\ - 0.00 \end{smallmatrix}$

Figure 6.2.1

BEARING TEST SPECIMEN

### 6.3 Instrumentation

- 6.3.1 An averaging type extensometer shall be used to measure the deformation of the bearing hole. It shall conform to the requirements given under tension tests, Section 1.

Per cent bearing strain is given in terms of percentages of bearing-hole diameter.

- 6.3.2 One set of extensometer knife edges shall be placed on the edge of the specimen in line with the edge of the bearing hole closest to the end of the specimen. The other set of knife edges will be located on the clevis.
- 6.3.3 Room temperature tests will be made at temperatures between 65 and 85F. Elevated temperature tests will be held to within the temperature limits required by Section 1.9. Proof of acceptable temperature distribution and control in the bearing hole region shall be provided.

### 6.4 Strain Rate

- 6.4.1 A crosshead speed of between .005 to 0.01 in/min shall be used to the 2 per cent offset yield strength and  $0.05 \pm .01$  inch per minute beyond the yield point to failure.

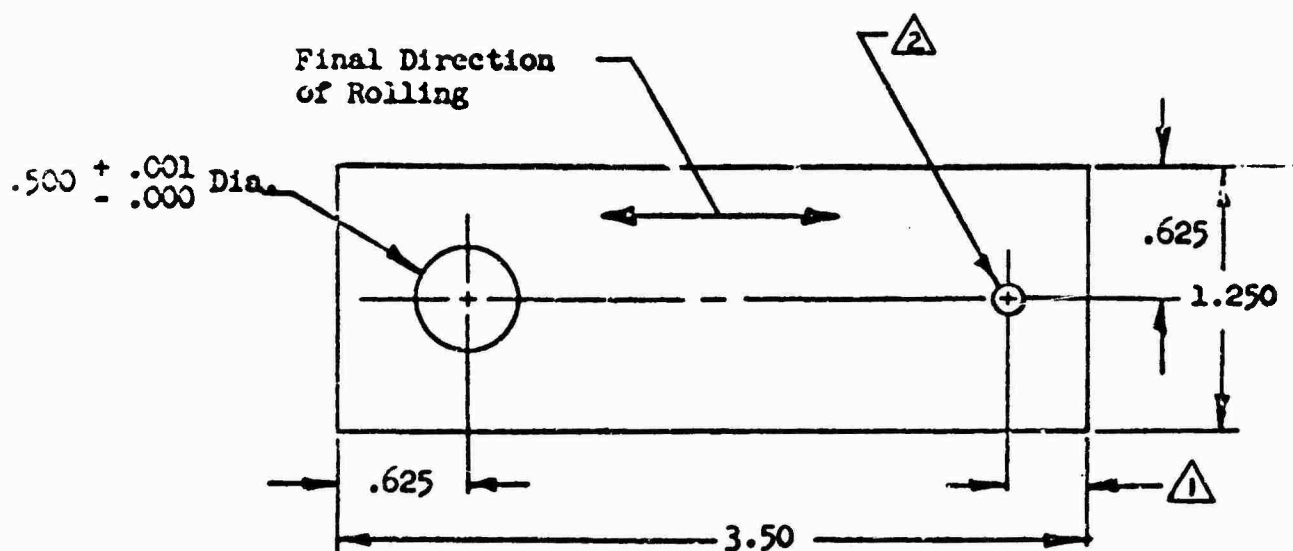
### 6.5 Test Data

- 6.5.1 Stress-strain curve shall be obtained to at least 3 per cent offset for determination of bearing yield strength at 2 per cent offset. Stress and strain shall be measured as indicated above. Test to be continued to the ultimate load to measure ultimate bearing strength.
- 6.5.2 Specimen thickness shall be measured at three points  $120^\circ$  apart at the bearing hole. Hole diameter shall be measured perpendicular to loading axis (center line of bearing and loading holes), to within 0.0005 inches.
- 6.5.3 All pertinent data shall be reported including the specimen orientation with respect to the rolling direction, test temperature, loading rate, and full details on chemical and metallurgical features of the material. In the case of elevated temperature tests, the results of post-test chemical analysis and metallographic inspection must be furnished.

### 6.6 References

J. R. Kattus and H. L. Lessley, "Determination of Compressive, Bearing, and Shear Creep of Sheet Metals," Proc. ASTM, Vol. 61, 1961, p. 920-930.

A. A. Moore and J. A. Gusack, "An Autographic Bearing-Strength Test and Typical Test Values on Some Magnesium Alloys at Room and Elevated Temperatures," Proc. ASTM, Vol. 56, 1956, p. 834-841.



NOTES:

1.  $0.250 \pm .005$  for 0.010, 0.012, and 0.018 thick specimens,  $0.375 \pm .005$  for 0.030 thick specimens.
2.  $0.1250 \pm .0005$  dia. for 0.010, 0.012, and 0.018 thick specimens,  $0.1875 \pm .0005$  dia. for 0.030 thick specimens.
3. Radius all edges  $1/2t$ , where  $t$  = material thickness, and corners 0.10 on specimens to be coated.

BEARING SPECIMEN CONFIGURATION

FIG 6.2.2

Third Report of Task Force on Uniform Procedures for Structural Design  
Data Collection of the Panel on Titanium Sheet Rolling Program, MAB 121-M.

7. Stress Rupture and Creep Testing

7.1 Specimen Dimensions

The requirements and recommendations of Sections 1.1 and 1.1.1 shall apply to the stress rupture and creep specimen.

7.2 Gripping Method - Optional

7.3 Surface Finish

The requirements of Section 1.3 shall apply.

7.4 Pretest Inspection

The requirements of Section 1.4 shall apply.

7.5 Loading Apparatus and Methods

The loading machine shall comply with the requirements of ASTM E-139. Calibration shall be performed in accordance with ASTM E-4. In addition, the following considerations should be especially noted:

Refractory metal creep tests must be conducted in controlled environments. There is no standard chamber or apparatus which has been certified or reviewed by any agency for this use. Thus, there are a variety of loading mechanisms and sealing techniques employed. For uniformity and comparison of data, each experimenter must be prepared to demonstrate by calibration data that the influence of friction, bellows, etc., are properly recognized and accounted for in calculating applied stresses. Until such time as more experience has been acquired, it is appropriate to require calibration of the test device under operating conditions at the lowest and highest loads required for the program.

7.6 Strain Measurement

In general, the requirements of ASTM E-139 shall be followed in making strain measurements. It is recognized that strain recording devices for tests in excess of 2000F are not readily available at the present time. Apparatus of differential transformer type has been developed which is capable of continuous strain recording to temperatures up to 3000F. Optical systems have also been developed which are useful to temperatures up to 4000F. In conducting creep tests, it is important that an accurate strain measuring system be employed and that the general requirements of ASTM E-139 be followed, particularly with respect to the determination of accuracy and sensitivity of the strain measurement system.

7.7 Heating to Test Temperature

Specimens shall be brought to temperature at a rate commensurate with the maintenance of a vacuum of  $1 \times 10^{-4}$  but with a minimum time of five minutes. Temperature gradients shall be adjusted when the test temperature is reached to comply with the requirements of Section 7.8 and the specimen shall be held at the test temperature for fifteen minutes before loading. Any deviation from the above shall be recorded with the test data.

7.8 Temperature Measurement and Control

7.8.1 Temperature measurement shall be in accordance with Section 1.9.2.

7.8.2 Three thermocouples of the appropriate material shall be fastened at equal distances along the gage length of the specimen and shielded with metal foil, preferably of the alloy base material. Special care should be taken to ensure that the couples do not exert force or drag on the specimen. The furnace shall be controlled from one of the thermocouples and the other two should be constantly recorded. Where optical or emission temperature controllers are used, equivalent procedures must be observed to ensure temperature uniformity over the gage length. Temperature measurement methods and calibration data must accompany test data. The indicated temperatures for all temperature sensors shall not vary by more than the following from the nominal test temperatures.

<u>Temperature Range</u>	<u>Maximum Permissible Variation</u>
To 1800F	$\pm 10^{\circ}$
Over 1800F up to 2800F	$\pm 20^{\circ}$
Over 2800F up to 3500F	$\pm 30^{\circ}$
Over 3500F up to 4000F	$\pm 35^{\circ}$

7.9 Test Environment

Testing shall be conducted under conditions to ensure that the surface of the specimen shall not be chemically affected during test. Vacuums of better than  $10^{-5}$  mm of mercury are recommended.

7.10 Post-Test Inspection

Following test, a representative specimen or specimens shall be subjected to the same tests as specified in Section 1.4. Any changes in chemical analysis, hardness, or structure shall be noted.

7.11 Reports

Test reports shall include all the pertinent data as required by the form shown in Appendix A. In addition, full information of pretest and post-test inspection will be reported and all details regarding test environment.

8. Weld Evaluation Test

8.1 Fusion Weld

8.1.1 Specimen Blanks

Sheet samples shall be butt welded to simulate accurately the welding technique and conditions. The sheet shall be fully representative of the product under evaluation. Welds will be made with the desired orientation and annealed or otherwise treated, after welding, as required by applicable material or process specifications.

8.1.2 Test Specimen

Test specimens shall be prepared with the weld joint bisecting the gage section at the center of the gage length. Specimen dimensions shall conform with Section 1.1 of this specification. If required, the weld bead may be ground or milled to smooth surface.

8.1.3 Test Method

The welded specimen shall be tested in accordance with the requirements of the applicable MAB test procedure.

8.2 Spot Weld Joint Efficiency in Shear

The spot weld joint efficiency in shear is influenced by the size, location, and metallurgical history of the weld nugget, as well as the thickness of the sheet. The test weld and specimen will, therefore, be standardized according to the practices recommended by the American Welding Society, Ref. 2.

8.2.1 Specimen Preparation

The specimen blanks, two required per sample, shall be cut from a flat sheet in the required orientation with the rolling direction. The blanks are to be machined and drilled to the dimension in Fig. 8.2.1. Care must be exercised to minimize eccentricity of the loading tabs, or pin holes. Any other suitable gripping method may be substituted.

Specimens shall be tested in the "as welded" and "after heat treatment condition," as desired.

Welding practice shall conform to that recommended in Ref. 2. Surface indentation shall be maintained to less than 2 per cent of sheet thickness.

8.2.2 Loading rate and testing machines shall conform to those for shear ultimate strength in Section 4.

### 8.3 Joint Efficiency

For the spot weld, the ultimate shear strength is the maximum shear load divided by the net shear area. The joint efficiency of the spot weld is defined as the ratio of the ultimate shear load divided by the product of the ultimate strength, (obtained in Section 1 for the same sheet material and the same orientation to the rolling direction), the specimen thickness,  $t$ , and the width,  $w$ , Fig. 8.2.1. The fracture shall be inspected to determine any irregularities influencing the test. The joint efficiency of the fusion weld is defined as the ratio of the ultimate strength of the welded specimen to the ultimate strength of the unwelded sheet.

### 8.4 Reports

All pertinent data shall be reported, including specimen orientation with respect to rolling direction, test temperature, loading rate, full details on welding technique, chemical and metallurgical features of the stock, and strength data. Temperature variations shall conform to limits given in Section 1. At least three readings of thickness and width of weld tab shall be determined before welding. The test report form shown in Appendix A is recommended.

### 8.5 References

- (1) J. Heuschkel, "The Expression of Spot-Weld Properties," Welding Journal, October, 1952, Vol. 31, pp 931-943.
- (2) American Welding Society, "Welding Handbook," Section 2, Recommended Practices, 4th Edition.

## 9. Delamination (Reverse Bend) Test

### 9.1 Scope

The method covers test procedures for a reverse bend test to evaluate the resistance of refractory metal and alloy sheet to delamination or splitting failures. It should be noted that results obtained by this method have not been correlated with susceptibility to delamination in sheet metal cutting, blanking, drilling, or other forming operations.

### 9.2 Equipment and Materials

#### 9.2.1 Testing Machine

Section 5.2.1 shall apply.

#### 9.2.2 Bend Fixture

Section 5.2.2 shall apply for the initial  $90^\circ$  and all subsequent forward  $90^\circ$  bends. For the reverse bend, the specimen shall be flattened





9.2.2 Bend Fixture (continued)

between two smooth finished plane parallel surfaces. All bearing surfaces should be lubricated if possible to reduce friction.

9.3 Test Specimens

Section 5.3 shall apply.

9.4 Test Procedures

9.4.1 Test Temperature

Tests shall be conducted at a temperature of 100F above the 4T or other specified bend transition temperature or at 65 to 85F if the bend transition temperature is below room temperature. Section 5.4.1 shall apply for temperature measurement and control.

9.4.2 Test Atmosphere

Section 5.4.2 shall apply.

9.4.3 Load Rate

Section 5.4.3 shall apply for the load rate in both forward and reverse bending.

9.4.4 Forward Bend

Section 5.4.3 shall apply to the initial forward and all subsequent forward bends.

9.4.5 Reverse Bend

The bent specimen shall be flattened to approximately its original shape by reverse bending. This shall be accomplished by compression of the bent specimen between two flat parallel surfaces. It is suggested that stops be used on the machine to prevent loading of the flattened specimen in compression. Springback angle after flattening shall be measured.

9.5 Test Results

9.5.1 Failure Criterion

The specimen shall be alternately forward and reverse bent (flattened) until failure occurs (Figure 3). Specimen edges shall be inspected after each reverse bend to determine time of failure. A delamination failure is defined as one in which a crack occurs in the plane of the sheet between the upper and lower surfaces, (Figure 3). The total number of 90° bends forward and reverse, before failure shall be taken

9.5.1 Failure Criterion (continued)

as the measure of delamination tendency. If failure occurs on the first reverse bend, the number of degrees of back bend before failure shall be estimated and reported.

9.6 Report Requirements

- a. Material - condition and history
- b. Test temperature, time of tests for each bend and rate of crosshead or ram travel.
- c. Springback angle on flattening.
- d. Total number of 90° bends, forward and reverse, including initial forward bend, to failure.
- e. Type of failure, if other than delamination, specify precise nature.

The recommended report form is shown in Appendix A.

10. Recrystallization Temperature Determination

10.1 Equipment and Materials

10.1.1 Equipment

Conventional diamond pyramid hardness (Vickers or equivalent), and metallographic equipment shall be used to measure extent of recrystallization.

10.1.2 Specimens

Samples of suitable size shall be cut from the "as-received" sheet. Surface condition shall not be changed in sample preparation.

10.2 Test Procedure

10.2.1 Annealing

All specimens shall be annealed one hour at temperature in vacuum or inert atmosphere of sufficient quality to prevent surface contamination. Specimens shall be heated to annealing temperatures in no less than 5 and no more than 60 minutes. Temperature shall be controlled to within  $\pm 10^\circ\text{F}$  of set temperature. Tests shall be made at 50F intervals to establish recrystallization temperature.

10.2.2 Estimate of Recrystallization

The extent of recrystallization shall be determined by metallographic examination of both transverse and longitudinal cross sections at 100X

10.2.2 Estimate of Recrystallization (continued)

magnification. The diamond pyramid hardness shall be measured on both surfaces with a minimum of five readings on each side.

10.2.3 Recrystallization Temperature

The recrystallization temperature shall be defined as the minimum temperature for which in one hour:

- a. The structure is at least 50 per cent recrystallized;
- b. The drop in hardness is at least  $2/3$  of the total drop from initial condition to the fully recrystallized condition.

10.3 Report Requirements

The following data shall be reported:

- 10.3.1 Recrystallization temperature.
- 10.3.2 Per cent structure recrystallized at one hour at recrystallization temperature.
- 10.3.3 As-rolled hardness.
- 10.3.4 Fully annealed hardness.
- 10.3.5 Per cent drop in hardness (per cent of total drop on full annealing) after one hour at recrystallization temperature.

11. Fatigue Test

Although there are no recognized standards for fatigue testing of sheet materials, simple bending fatigue tests may be made by several methods based upon magnetic, mechanical, or pneumatic drives. Fatigue data on refractory metal sheet must be accompanied by a complete description of test methods, test conditions, and material history. In addition, limits of accuracy of all instrumentation should be clearly stated and supporting data provided. A report form similar to that shown in Appendix A is recommended.

12. Thermal Conductivity Test

Thermal conductivity measurements are exceedingly difficult and expensive to obtain with a high degree of precision. In general, two types of approach have been used. One is very complicated experimentally and the data are easily analyzed. The steam calorimeter technique described in ASTM is an example of this approach. There are many experimental difficulties involved in trying to utilize such an apparatus for refractory metals at high temperatures in controlled environments. The

## 12. Thermal Conductivity Test (continued)

method described herein involves the use of rather simple experimental techniques to synthesize the data and is probably the cheapest and simplest procedure available. Its most serious drawback is the errors introduced when alloying constituents of 10 per cent or more are present in the base metal. If alternate methods are employed, they should be fully described.

### 12.1 Scope

This procedure shall apply to determination of thermal conductivity of refractory metal and alloy sheet.

### 12.2 Equipment

It is recommended that the thermal conductivity be derived from the electrical resistivity by the formula:

$$K = 0.57 \times 10^{-8} P T + 0.03$$

Where K is measured in calories cm/cm<sup>2</sup>sec<sup>0</sup>c, P in reciprocal ohm-cm units, and T is absolute temperature <sup>0</sup>K. The constant  $0.57 \times 10^{-8}$  is a theoretical value and is subject to some uncertainty. The choice of a different value for K, should be explained. A furnace shall be used capable of maintaining a temperature gradient over the specimen of not greater than  $\pm 10^{\circ}\text{F}$  up to  $1800^{\circ}\text{F}$ ,  $\pm 20^{\circ}\text{F}$  above  $1800$  to  $2800^{\circ}\text{F}$ ,  $\pm 30^{\circ}\text{F}$  between  $2800$  to  $3500^{\circ}\text{F}$ , and  $\pm 35^{\circ}\text{F}$  above  $3500^{\circ}\text{F}$  up to and including  $4008^{\circ}\text{F}$ . A protective environment sufficient to prevent deleterious contamination shall be maintained during test. The change in resistance of the specimen shall be measured by an apparatus capable of determining such changes with an accuracy of  $\pm 1$  per cent or better. A Kelvin double bridge is suitable for this purpose.

### 12.3 Specimens

A one-quarter inch strip of the sheet thickness six or more inches long shall be used for the specimen. The strip shall be spiraled in a helix and supported on a ceramic rod that is free of outgassing at the pressures and temperatures used in the test. All high-temperature electrical connection inside the furnace shall be of the same material as the specimen.

### 12.4 Test Procedure

The specimen shall be brought to test temperature in no less than five minutes or no more than sixty minutes, and held at test temperature for fifteen minutes before the resistance readings are taken. The measurements shall be made in such a way that the effects of thermoelectromotive force and parasitic currents are avoided. Measurements shall be made at a sufficient number of temperatures to determine the

#### 12.4 Test Procedure (continued)

characteristic of the material. For plotting a curve six or more observations are generally made; the temperature of the measuring apparatus shall be noted at frequent intervals during the test of each specimen. Room temperature resistance should be measured both before and after test.

#### 12.5 Report

The report shall include the following:

- a. Identification of specimen.
- b. Description of material.
- c. Specimen thickness and width to the nearest  $\pm 0.5$  per cent.
- d. Approximate resistance and distance between potential terminals to the nearest  $\pm 0.5$  per cent.
- e. Tabular list of resistance or changes in resistances and temperatures in order taken.
- f. Temperature of measuring apparatus and room at start and finish of the test.
- g. Data shall be provided to demonstrate the accuracy of the temperature sensor device.
- h. Data shall be provided to demonstrate the accuracy of the resistance measuring equipment.

A report form similar to the form shown in Appendix A is recommended.

#### 12.6 Reference

Electrical Resistivity of Commercially Pure Titanium as a Function of Temperature, NEPA 1826, April 16, 1951.

#### 13. Thermal Expansion Test

##### 13.1 Scope

This procedure shall apply to determination of thermal expansion and phase changes of refractory metal and alloy sheet.

##### 13.2 Equipment

A chamber in which a suitable protective environment can be achieved, a furnace capable of heating the specimen to the desired temperature, thermocouples, and a length measuring device are the minimum requirements.

### 13.3 Specimens

The specimen should be prepared so as to ensure accurate measurements. The specimen should not be subject to curling, buckling, or bowing during test.

### 13.4 Methods

Since the Dilatometric Analysis is intended to determine phase changes as well as thermal expansion, the type of equipment used should give a continuous record of expansion, with a simultaneous record of temperature. An extension rod dilatometer, with recording by the optical lever or variable transformer, would give this type of record. If the specimen holder and push rod are of fused quartz, this equipment will be usable to 1800F. By using sapphire parts, its temperature range can be extended to 3275F. For temperatures higher than this, the actual measurement of specimen length by micrometer-telemicroscope is most suitable. This method, however, does not give a continuous record of expansion. These methods of measurement require a rod shaped specimen. They are capable of measuring expansions of  $10^{-5}$  in/in up to 1800F and  $10^{-4}$  in/in above that.

For precision tests an interferometer or other techniques sensitive to  $1 \times 10^{-6}$  inches should be utilized for length measurements produced by phase changes or when very precise thermal expansion data are required. This method is generally limited to temperatures of approximately 1800F. Measurements of expansion in the thickness direction could be accomplished by this method.

Qualitative testing may be accomplished by simple dial gages or similar instruments sensitive to  $1 \times 10^{-3}$  inches on specimens 6 to 10 inches in length when comparative test data are desired. Because of the inherent geometric instability of sheet specimens, it is suggested that the test procedure impose a slight tensile rather than a compressive load on the material. No permanent deformation should be allowed to occur as a result of these loads.

### 13.5 Test Procedures

ASTM E 80-49T should be followed.

### 13.6 Report Requirements

Recommendations of ASTM E 80-49T should be followed and complete information should be furnished, as indicated by the report form shown in Appendix A.

14.     Specific Heat

14.1    Scope

This procedure shall apply to determination of specific heat for refractory metal and alloy sheet by the drop calorimeter method.

14.2    Equipment

The furnace and temperature control instrumentation shall be capable of meeting the requirements of Section 1.9.1. The calorimeter shall be adiabatic with provisions for measuring changes of temperature 0.01°F.

14.3    Specimens

Any suitable test coupon for the proper equipment shall be used.

14.4    Test Procedure

14.4.1   Drop Calorimeter Method

Specimens shall be in a protective environment or suitably encapsulated to prevent contamination. Specimens shall be heated to test temperature in no less than 5 and no more than 60 minutes. The sample shall be dropped into a close fitting closed thin tube of similar metal. A gate valve shall separate the furnace and calorimeter section of the tube.

14.4.2   Other Methods

The pulse-heating method of Pallister could be coupled with the electrical resistance measurement to reduce the needed initial expenditure<sup>1</sup>. The U.T. adiabatic method is also suggested<sup>2</sup>.

14.5    Reports

Three drops at each specified temperature shall be made. The enthalpy shall be reported in BTU/lb.

Complete material and test procedure information should be furnished on a form similar to that shown in Appendix A.

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<sup>1</sup> P. R. Pallister, "Specific Heat and Resistivity of Mild Steel," Journal of Iron and Steel Institute, April 1957, p. 474.

<sup>2</sup> E. E. Stansbury, et.al., "Adiabatic Calorimetry for Metals in the Range 50 - 1000C," Review of Scien. Instr. (30), p. 121, 1959.



15. Modulus of Elasticity Determination

15.1 Dynamic Modulus

15.1.1 Scope

This procedure is suggested for the determination of modulus of elasticity of refractory metal and alloy sheet by the dynamic method. Other methods may also be used. In any case a complete description of test apparatus and methods should accompany test data.

15.1.2 Equipment

The testing apparatus shall consist of the following:

- a. Driving circuit. The driving circuit shall consist of a variable frequency audio oscillator, an amplifier, and a driving unit. The oscillator shall be calibrated to read within  $\pm 2$  per cent of the true frequency over the range of use (100 to 10,000 cycles per second). The combined oscillator and amplifier shall be capable of delivering at least 5 watts power output with not more than 5 per cent distortion and shall be provided with a means of controlling the output. The oscillator and amplifier shall be capable of producing a voltage that does not vary more than  $\pm 20$  per cent over the frequency range and, in combination with the driving unit shall be free from spurious resonances that will be reflected in the output. The use of a frequency band trap is recommended.
- b. Pickup Circuit. The pickup circuit shall consist of a pickup unit, an amplifier, and an indicator. The pickup unit shall generate a voltage proportional to the amplitude, velocity, or acceleration of the test specimen. Either a piezoelectric or magnetic pickup unit meeting these requirements may be used. The amplifier shall have a controllable output of sufficient magnitude to actuate the indicator. The indicator shall consist of a voltmeter, milliammeter or cathode ray oscilloscope. The use of an oscilloscope as an indicator may be necessary when specimens are to be tested for which the fundamental frequency range is unpredictable. The oscilloscope is valuable also for checking the equipment for distortion and drift. The response of the pickup unit shall be proportional to the motion of the test specimen in accordance with the characteristic of the type of pickup selected, and shall be free from spurious resonances in the normal operating range.
- c. Specimen Support. Since tests may be required to 4000F, resistance or induction furnace will be used in vacuum of  $10^{-5}$  torr or an equivalent protective environment. The specimen shall be supported on wire stirrups near nodal points attached to the driving and pickup units. This allows the driving and pickup units to be placed outside of the furnace. The high vacuum may reduce the use of some driving and pickup units. This allows the specimen to vibrate without restriction in a free-free transverse mode.

#### 15.1.3 Test Specimens

The test specimen shall be a quarter-inch wide strip, four-inch long of sheet thickness.

#### 15.1.4 Test Procedure

The specimen shall be placed in the stirrups in such a manner that it may vibrate without restriction in a free-free transverse mode. The specimen shall be forced to vibrate at varying frequencies. At the same time, the indication of the amplified output of the pickup shall be observed. The frequency of the test specimen that results in a maximum indication having a well-defined peak on the indicator, and at which observation of nodal points indicates fundamental transverse vibration shall be recorded as the fundamental transverse frequency.

#### 15.1.5 Test Calculations

Young's modulus of elasticity shall be calculated from the fundamental transverse frequency, weight, and dimensions of the test specimen as follows:

$$E = 0.5409 \times 10^{-5} \frac{WL^3 f^2}{bh^3} \text{ lbs/in}^2 \text{ units in inches and grams.}$$

Where:

E = Young modulus of elasticity in pounds per square inch.

W = Weight of specimens in grams.

b = Width of specimen in inches.

h = Thickness of specimen in inches.

$f^2$  = Fundamental frequency, cycles per second squared.

L = Length of specimen in inches.

#### 15.1.6 Test Reports

Complete material and test methods information shall be furnished on a report form similar to that shown in Appendix A.

#### 15.2 Static Modulus

The accurate determination of the modulus of elasticity through static-type tests is difficult, even for conventional alloys. The problem is rendered more difficult when sheet specimens are utilized, protective environments are required, and temperatures in excess of 1800F are desired. A comparison of modulus data shows that static values

15.2 Static Modulus (continued)

generally are only one-half to one-fifth of those obtained by dynamic techniques. Most of this deviation is attributable to experimental error. A number of experimental techniques were considered which conceivably could improve the accuracy of static tests, but no proven procedure appears wholly satisfactory at this time. The committee feels that it may be preferable not to suggest a standard method, but to request that each experimenter report in detail how the test was performed. At some later date, these procedures could be reviewed to determine which provide the best results.

Whatever technique is used, the same general procedure could be followed for controlling temperature and environment as specified for tensile tests. In addition, strain should be measured to at least  $1 \times 10^{-4}$  in/in and stress to within 1.0 per cent.

APPENDIX A

General Recommendations

on

Test Reports

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The attached test report form is recommended. This form requires information on materials history and test methods essential to a realistic evaluation of the test data. The presentation of complete material and test information should help to prevent erroneous conclusions or avoid invalid comparisons of data.

TEST REPORT FORM

REFRACTORY METAL SHEET EVALUATION

---

MATERIAL \_\_\_\_\_ HEAT OR MELT NO. \_\_\_\_\_

PRODUCER \_\_\_\_\_ WIDTH \_\_\_\_\_ THICKNESS \_\_\_\_\_

MANUFACTURING DATA (Pertinent data regarding major manufacturing steps including reduction schedules and temperatures.)

CHEMICAL ANALYSES (Include interstitials.)

Pre test (Sheet sample) -

Post test (Specimen) -

METALLOGRAPHIC ANALYSES (Include hardness data and notes on surface effects.)

Pre test -

Post test -

SPECIMEN PREPARATION (Methods, Finish, etc.)

TEST DESCRIPTION

Specimen

Orientation

Test Temp.

Heating Method

Soak Time

Temp. Control

Test Machine

Test Environment

TEST PROCEDURE (Details on strain rates, strain measurement, temperature control, etc., as applicable, pertinent information on calibration of instruments, limits of accuracy, test environment, and all other information which may contribute to a more useful evaluation of the data.)

TEST REPORT FORM (continued)

REFRACTORY METAL SHEET EVALUATION

TEST RESULTS (Specific test data with complete information on test parameters.)

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10 AVAILABILITY/LIMITATION NOTICES		
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11 SUPPLEMENTARY NOTES		12 SPONSORING MILITARY ACTIVITY
13 ABSTRACT		
<p>This is the third revision of recommended test methods for refractory metal sheet published by the Materials Advisory Board Refractory Metals Sheet Rolling Panel. The Panel's objective was to provide a standard basis for the test evaluation of sheet materials produced under various Department of Defense development programs. A subpanel was appointed for this purpose. After a thorough canvas of prominent suppliers and users of refractory sheet metals, the first recommendations were published in September 1961 as MAB-176-M to define common tensile, creep-rupture, bend, and thermal properties tests. Subsequently, as the Department of Defense programs progressed, more specialized tests became necessary to evaluate fabrication characteristics and to provide preliminary design data. Again, after discussion with principal organizations engaged in direct applications, a revised edition was published April 22, 1963, as MAB-192-M. Since that time, criticisms and comments have been reviewed by the subpanel with qualified individuals representing governmental and commercial laboratories. The present edition is based upon these reviews. Changes have been made in the tests described in Sections 1 through 6. The tests in Sections 7 through 15 are unchanged from Report 192-M.</p> <p>In preparing these recommendations, the subpanel has deferred where possible to prominent ASTM standards. The subpanel thanks the many government and industrial laboratories who gave valuable advice and comment.</p>		

## Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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